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(19)

(54) IMPROVEMENTS IN OR RELATING TO THE COMMINUTION OF SOLID MATERIALS

(71) We, ENGLISH CLAYS LOVERING POCHIN & COMPANY LIMITED, a British Company, of John Keay House, St. Austell, Cornwall, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the comminution of solid materials and, more particularly but not exclusively, is concerned with the comminution of solid materials comprising calcium carbonate in order to provide fine particulate calcium carbonate suitable for use as a pigment or filler material.

Conventionally the harder types of calcium carbonate-containing mineral, such as calcite marble, vein calcite, dolomite and limestone, are quarried by blasting and the large lumps of rock thus liberated are broken down in heavy duty crushers. The final product of the crushing plant is generally in the form of chippings having a maximum particular size of about 20 mm. These chippings are then further reduced in size by milling in a ball mill charged with a suitable grinding medium which may conveniently be flint pebbles having diameters of about 50-100 mm although other grinding media may equally well be used. The ball milling step may be performed wet or dry but when the product is required to be comminuted further it is preferred to grind the chippings in the form of an aqueous slurry.

The ball mills conventionally used have the disadvantages that their capital cost is high and that the amount of energy which can be brought to bear on the feed material is governed by the weight of the grinding medium and the maximum distance through which they can fall under gravity which is generally a little less than the internal diameter of the mill. For this reason the grinding efficiency of a ball mill, measured in terms of the weight of feed per hour which can be reduced to a given particle size, tends to be rather low. In addition, the cost of maintaining a conventional ball mill may be high because it is supported at each end in heavy roller or ball bearings which are subject to wear and must be renovated at frequent intervals. Also, while grinding the feed material and grinding balls or pebbles themselves become abraded or fractured to some extent and the product is therefore contaminated with the material of which the pebbles or balls are constituted.

According to the present invention there is provided a process, for comminuting a solid material substantially all of which consists of pieces not larger than about 20 mm, which comprises charging the grinding chamber of an attrition grinding mill with a slurry of said solid material, water and a dispersing agent, said grinding chamber being provided with an internal rotatable impeller for agitating said slurry, agitating said slurry in said grinding chamber to comminute said solid material to the desired particle size; continuously withdrawing from the grinding chamber a part of said slurry containing comminuted pieces of said solid material; and feeding to said chamber further quantities of said solid material, water and dispersing agent; wherein there is present in said grinding chamber during the agitation of said slurry no particulate solid other than material derived from said solid material, wherein the rates of feeding said water, said dispersing agent and said solid material to said grinding chamber are controlled so that the part of said slurry containing comminuted solid material which is

withdrawn from the grinding chamber contains at least 50% by weight of solids, and wherein the volume of slurry in said grinding chamber is maintained substantially constant.

Advantageously, the internal rotatable impeller is driven by an electric motor, the current drawn by the electric motor being maintained between predetermined upper and lower limits. Preferably, the rates of feeding to said grinding chamber said water, said dispersing agent and said solid material are controlled so that the current drawn by the electric motor driving said internal rotatable impeller can be maintained at not less than about 80% of its full load current.

The process of the invention will now be described by way of example by reference to its application to the comminution of solid materials comprising calcium carbonate.

The solid material initially charged to said grinding chamber and that subsequently fed to the grinding chamber may be prepared from a single batch of calcium carbonate obtained from a single source or it may be prepared from separate batches of calcium carbonate obtained from one or several sources. If necessary, any large lumps of the solid material are broken before being supplied to the grinding chamber to ensure that substantially all of the solid material in the grinding chamber consists of pieces not larger than 20 mm. Each batch of solid material may be crushed to a different extent to other batches, or the particle size distribution of each batch of solid material may be modified by sieving, to give batches of calcium carbonate which can be combined together in any proportions to give a blend which is particularly advantageous, for example as regards particle size distribution. The calcium carbonate can be obtained from any source, but the process of the invention is of particular value for comminuting hard varieties of calcium carbonate containing mineral, including one or more of calcite marble, vein calcite, dolomite and limestone.

The dispersing agent used in the process of the invention may be a water-soluble salt of a polysilicic acid, a water-soluble salt of a polyacrylic or a polymethacrylic acid having a number average molecular weight not greater than 5,000 or a copolymeric dispersing agent of the type disclosed in British Patent Specification No. 1,414,964. The amount of dispersing agent used will generally be in the range of from 0.1% to 0.6% by weight based on the weight of dry solid material. The solids content of the slurry in the grinding chamber is preferably such that the slurry withdrawn from the grinding chamber has a solids content of at least 60% by weight but not greater than 80% by weight.

The agitation of the slurry in the grinding chamber is preferably carried out under conditions such that the amount of energy dissipated in the slurry is at least 30 horsepower hours per ton of dry solid material; but it will not normally be more than 250 horsepower hours per ton (80-659 $kJkg^{-1}$). The attrition grinding mill may be of the type described in British Patent Specification No. 1,469,028 which includes an outlet means across which is disposed s sieve which retains coarser particles and allows a slurry of finer particles to pass therethrough. The aperture size of the sieve is conveniently in the range from 0.1 to 0.5 mm (between No. 150 mesh and No. 30 mesh B.S. sieve).

After the slurry of comminuted solid material has been withdrawn from the grinding chamber, the solid material may be further comminuted by agitation in the grinding chamber of a second attrition grinding mill, the grinding chamber being charged with a particulate grinding medium consisting of particles having sizes in the range from 0.15 mm to 2 mm. The amounts of particulate grinding medium and slurry in the second attrition grinding mill are preferably such that the volume ratio of particulate grinding medium to slurry in the grinding chamber is in the range 0.5:1 to 1.5:1, more preferably in the range 0.9:1 to 1.1:1. An especially suitable material for use as the grinding medium is silica sand.

There will now be described, by way of example and with reference to the accompanying drawing, one embodiment of apparatus suitable for carrying out the process of the invention.

An attrition grinding mill includes a grinding chamber 1 which is octagonal in cross section and has a width which is a little smaller than its height. At is lower end it is provided with an outlet box 2 with a sieve 3 which permits a slurry of sufficiently ground particles to pass therethrough while retaining particles which have not been sufficiently finely ground. A feed chute 4 is provided to enable solid particulate calcium carbonate mineral to be fed to the grinding chamber 1. Conduits 5 and 6, provided with control valves 7 and 8 respectively, serve to introduce water and dispersing agent respectively at controlled rates into the grinding chamber 1. The calcium carbonate mineral to be ground is deposited on an endless belt conveyor 9 at the position indicated by the arrow 10 and is conveyed to the grinding chamber and discharged thereinto via the feed chute 4.

The grinding chamber is provided with an impeller 11 which comprises eight straight round bars 12 mounted on a central vertical shaft 13 which is driven by an electric motor 14 through a gear box 15. Electric power is supplied to the motor through a cable 16 and the electric current flowing in this cable is sensed by a measuring unit 17 which transmits a signal proportional to the current drawn by the electric motor 14 to a control unit 18 which controls an electric motor 19 which

drives the endless belt conveyor 9. When the current drawn by the electric motor 14 exceeds a certain first predetermined value the motor 19 is de-energised by the control unit 18 and the supply of calcium carbonate mineral to the grinding vessel is halted. When the current drawn by the electric motor 14 falls again below a second predetermined value the control unit 18 re-energises the motor 19 and the supply of calcium carbonate mineral is restarted.

The control unit 18 is similar to that described in British Patent Specification No. 1, 469, 028 and is provided with a first preset potentiometer which determines the upper limit of the current drawn by the motor 14 at which the motor 19 is de-energised and a second preset potentiometer which determines the differential between the upper and lower limits. The level of liquid in the grinding chamber is maintained constant by connecting to the lowest point of the outlet box a conduit 20 which is provided with an inverted U-shaped portion 21, the highest point of the inverted U being at the desired liquid level. A vent 22 is provided at this point as a siphon breaker. Ground slurry passes through conduit 20 to a product sump 23.

This invention is further illustrated by the following examples.

EXAMPLE 1

Large lumps of Carrara marble were broken by conventional crushing equipment and after large particles had been removed by sieving the crushed material had the particle size distribution shown in Table 1*a*.

TABLE 1*a*

Size range: mm	*% by weight in size range*
−1.0 + 0.6	3.4
−0.6 + 0.315	14.7
−0.315 + 0.25	9.1
−0.25 + 0.053	50.5
−0.053	22.3
	100.0

The crushed material was mixed with water, to form a slurry containing 65% by weight of dry solids, and with 0.48% by weight, based on the weight of dry solids, of a sodium polyacrylate dispersing agent having a number average molecular weight of 1650.

The resultant slurry was fed at the rate of 3.52 tonnes per hour of dry solids to the grinding chamber of an attrition grinding mill of the type described in British Patent Specification No. 1,469,028 which was provided at the bottom with a sieve having apertures of width 0.25 mm which allowed a slurry of finely ground particles to pass, but retained the coarser particles. At the same time there were fed to the attrition grinding mill, at the rate of 1.01 tonnes per hour, marble chippings which were produced by breaking Carrara marble with conventional crushing equipment and which had the particle size distribution shown in Table 1*b* below:—

TABLE 1*b*

Size range: mm	*% by weight in size range*
−12 + 6	21.5
−6 + 4	54.0
−4 + 2	24.3
−2 + 1	0.2
	100.0

The contents of the grinding mill were agitated by means of the impeller. No additional grinding medium was introduced to the grinding chamber and there was dissipated in the suspension 47 horsepower hours of energy per ton or dry calcium carbonate (124 $kJkg^{-1}$). The slurry of finely ground particles passing through the sieve was sampled at intervals and the samples were combined to make an overall sample representative of the total product of the run which continued for 4 hours 40 minutes. The ground product had a particle size distribution such that 35% by weight consisted of particles having an equivalent spherical diameter smaller than 2 microns, 23% by weight consisted of particles having an equivalent spherical diameter larger than 10 microns and 7.6% by weight consisted of particles which were retained on a No. 300 mesh B.S. sieve (nominal aperture 53 microns).

EXAMPLE 2

Large lumps of Carrara marble were broken by conventional crushing equipment and the crushed material had the particle size distribution shown in Table 2 below:—

TABLE 2

Size range: mm	*% by weight in size range*
−12 + 6	61.8
−6 + 4	21.9
−4 + 2	13.5
−2 + 1	1.0
−1	1.8
	100.0

The crushed material was fed into the attrition grinding mill which was used in Example 1 at the rate of 3.72 tonnes per hour together with water at the rate of 2,260 litres per hour and the sodium polyacrylate dispersing agent which was used in Example 1 at the rate of 0.19% by weight, based on the weight of dry solids. The contents of the grinding mill were agitated by means of the impeller. No additional grinding medium was introduced to

the grinding chamber and there were dissipated in the suspension 65 horsepower hours of energy per ton of dry calcium carbonate (175 $kJkg^{-1}$). The slurry of finely ground particles passing through the sieve was sampled at intervals and a bulk sample representative of the total output of ground material was prepared. The product consisted of a deflocculated slurry containing 62.2% by weight of ground calcium carbonate having a particle size distribution such that 26% by weight consisted of particles having an equivalent spherical diameter smaller than 2 microns, 39% by weight consisted of particles having an equivalent spherical diameter larger than 10 microns and 2.3% by weight consisted of particles which were retained on a No. 300 mesh B.S. sieve.

The product slurry was passed through a sieve having an aperture of 0.120 mm to the grinding chamber of a second attrition grinding mill identical to the first but containing as grinding medium 2.5 tonnes of silica sand having particles in the size range 0.5 – 1.0 mm, the quantity of sand being such that the volume occupied by the sand was approximately equal to the volume occupied by the slurry in the grinding mill. Additional sodium polyacrylate dispersing agent was mixed with the slurry to raise the total amount of dispersing agent to 0.45% by weight based on the weight of dry calcium carbonate, and the slurry was passed continuously through the grinding mill, the contents being agitated by means of the impeller for a total time of 5 hours 16 minutes. The slurry of finely ground particles passing through the sieve was sampled at intervals and a bulk sample representative of the total output of ground material was prepared. The product consisted of a deflocculated suspension containing 70.2% by weight of ground calcium carbonate having a particle size distribution such that 94% by weight consisted of particles having an equivalent spherical diameter smaller than 2 microns and 2% by weight consisted of particles having an equivalent spherical diameter larger than 10 microns. The autogenous grinding process according to the invention can therefore be seen to provide a suitable preliminary step to a conventional attrition grinding step for producing ultra-fine natural calcium carbonate.

EXAMPLE 3 (comparison)

As a comparison the crushed marble which was used as the feed material in Example 2 was ground to approximately the same particle size distribution as in Example 2 (namely 28% by weight having an equivalent spherical diameter smaller than 2 microns, 42% by weight having an equivalent spherical diameter larger than 10 microns and 1.3% by weight retained on a No. 300 mesh sieve B.S.) in a conventional pebble mill having a grinding charge of flint pebbles. The pebble mill could grind a batch comprising 11 tonnes of crushed marble but the time taken to reduce it to the desired degree of fineness was found to be 6 hours with a time of 1 hour for discharging and reloading. The average rate of production was therefore 1.57 tonnes of dry ground calcium carbonate per hour compared with 3.72 tonnes per hour produced by the process of the invention. One attrition grinding mill used in accordance with the process of the invention is therefore equivalent to two or three pebble mills used in accordance with a conventional process.

EXAMPLE 4

Large lumps of a white limestone were broken by conventional crushing equipment and the crushed material further comminuted in a hammer mill to give a material having the particle size distribution shown in Table 4 below:—

TABLE 4

Size Range (mm)	*% by weight in size range*
+12	7.9
–12 + 6	35.1
–6 + 4	11.6
–4 + 2	19.9
–2 + 1	10.3
–1	15.2
	100.0

The hammer milled material was fed into the grinding chamber of the attrition grinding maill shown in the accompanying drawing together with water at the rate of 948 litres per hour and the sodium polyacrylate dispersing agent used in Example 1 at the rate of 0.45% by weight, based on the weight of dry solids, to form a slurry containing at least 50% by weight of solids. The average throughput rate of the milled material was found to be 1.84 tonnes per hour but the endless belt conveyor 9 was operated at a speed such that the rate of delivery of the milled material to the feed chute 4 was greater than 1.84 tonnes per hour so that the current drawn by the electric motor 14 could be maintained at about 80% of its full load rating by energising and de-energising the conveyor motor 19 by means of the control unit 18. The contents of the grinding chamber 1 were agitated by means of the impeller. No additional grinding medium was introduced, and there were dissipated in the slurry 135 horsepower hours of energy per ton of dry calcium carbonate (357 $KJ.kg^{-1}$). The slurry of finely ground particles passing through the screen 3, which had an aperture size 0.250 mm was sampled at intervals and a bulk sample representative of the total output of ground material was prepared. The product consisted of a deflocculated slurry containing 65.7% by weight of ground calcium carbonate having a particle

size distribution such that 39% by weight consisted of particles having an equivalent spherical diameter smaller than 2 microns, 15% by weight of particles having an equivalent spherical diameter larger than 10 microns and 0.91% by weight of particles which were retained on a No. 33 mesh B.S. sieve.

The product was suitable for further grinding to form an ultra-fine calcium carbonate by the process described in Example 2 except that it was unnecessary to add a further quantity of the dispersing agent since sufficient had been added in the first attrition grinding stage.

These results show that, although the throughput rate is lower and the energy consumption higher than in the case of marble, the process of the invention is suitable for comminuting a hard calcium carbonate material such as limestone.

WHAT WE CLAIM IS:—

1. A process, for comminuting a solid material substantially all of which consists of pieces not larger than about 20 mm, which comprises charging the grinding chamber of an attrition grinding mill with a slurry of said solid material, water and a dispersing agent, said grinding chamber being provided with an internal rotatable impeller for agitating said slurry; agitating said slurry in said grinding chamber to comminute said solid material to the desired particle size; continuously withdrawing from the grinding chamber a part of said slurry containing comminuted pieces of said solid material; and feeding to said chamber further quantities of said solid material, water and dispersing agent; wherein there is present in said grinding chamber during the agitation of said slurry no particulate solid other than material derived from said solid material, wherein the rates of feeding said water, said dispersing agent and said solid material to said grinding chamber are controlled so that the part of said slurry containing comminuted solid material which is withdrawn from the grinding chamber contains at least 50% by weight of solids, and wherein the volume of slurry in said grinding chamber is maintained substantially constant.

2. A process according to Claim 1, wherein said internal rotatable impeller is driven by an electric motor and wherein the current drawn by the electric motor driving said internal rotatable impeller is maintained between predetermined upper and lower limits.

3. A process according to Claim 2, wherein the rates of feeding to said grinding chamber said water, said dispersing agent and said solid material are controlled so that the current drawn by the electric motor driving said internal rotatable impeller can be maintained at not less than about 80% of its full load current.

4. A process according to Claim 1, 2 or 3, wherein the solid material comprises calcium carbonate.

5. A process according to Claim 4, wherein said solid material comprises a hard variety of a calcium carbonate containing mineral.

6. A process according to Claim 4 or 5 wherein said solid material includes one or more of calcite marble, vein calcite, dolomite and limestone.

7. A process according to claim 4, 5 or 6, wherein the dispersing agent is a water-soluble salt of a polyacrylic acid or of a polymethacrylic acid, having a number average molecular weight not greater than 5,000.

8. A process according to Claim 7, wherein the amount of dispersing agent used is in the range of from 0.1% to 0.6% by weight based on dry solid material.

9. A process according to any one of Claims 4 to 8, wherein the rates of feeding said water, said dispersing agent and said solid material to said grinding chamber are such that the solids content of the slurry withdrawn from the grinding chamber is in the range of from 60% by weight to 80% by weight.

10. A process according to any one of Claims 4 to 9, wherein the amount of energy dissipated in the slurry in the grinding chamber during the agitation thereof is in the range of from 30 horsepower hours per ton of dry solid material to 250 horsepower hours per ton of dry solid material.

11. A process according to any one of Claims 4 to 10, wherein the slurry of solid material which is withdrawn from the grinding chamber is fed to a second attrition grinding mill in which the solid material is further comminuted by agitation in the grinding chamber of the second attrition grinding mill, the grinding chamber of the second attrition grinding mill being charged with said slurry and with a particulate grinding medium consisting of particles having sizes in the range of from 0.15 mm to 2.0 mm and the amounts of particulate grinding medium and said slurry charged to the grinding chamber of the second attrition grinding mill being such that the volume ratio of particulate grinding medium to said slurry in the grinding chamber is in the range of from 0.5:1 to 1.5:1.

12. A process, according to Claim 1, for comminuting a calcium carbonate-containing mineral substantially as described in any one of the foregoing Examples.

13. A process, according to claim 1, for comminuting a solid material substantially as hereinbefore described with reference to the accompanying drawings.

14. An apparatus for carrying out the process claimed in Claim 1, substantially as hereinbefore described with reference to, and as shown in, the accompanying drawing.

15. A solid material whenever comminuted by the process claimed in any one of claims 1 to 13.

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1569969 COMPLETE SPECIFICATION

1 SHEET *This drawing is a reproduction of the Original on a reduced scale*